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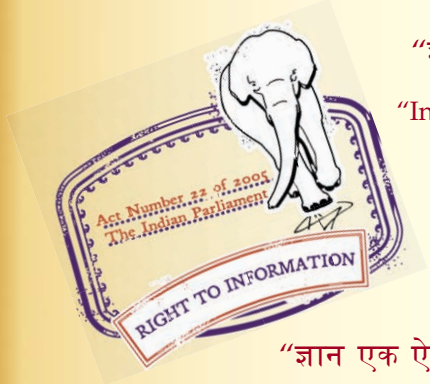

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Indian Standard

SPECIFICATION FOR AMYL ALCOHOL

(Revised)

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr. 4

June 1964

Indian Standard

SPECIFICATION FOR AMYL ALCOHOL

(Revised)

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Indian Standard

SPECIFICATION FOR AMYL ALCOHOL

(Revised)

0. FOREWORD

0.1 This revised Indian Standard was adopted by the Indian Standards Institution on 7 January 1964, after the draft finalized by the Alcohols and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1953. It was felt necessary to revise this standard in the light of present-day requirements. In the revised standard, a new grade suitable for testing milk has been added. Mention of the source of the material and the requirement for flash point have been deleted, and the requirement for the distillation range has been modified ensuring improved quality of material now generally available in the country. The new grade is based on the requirements of amyl alcohol prescribed in IS : 1224-1958 Determination of Fat in Whole Milk, Evaporated (Unsweetened) Milk, Separated Milk, Skim Milk, Buttermilk and Cream by the Gerber Method. A test for determining suitability of this grade of the material used for milk analysis has accordingly been added.

0.3 Wherever a reference to any Indian Standard appears in this specification, it shall be taken as a reference to the latest version of the standard.

0.4 This standard is one of a series of Indian Standards on alcohols and allied products.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960 Rules for Rounding Off Numerical Values (*Revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

0.6 This standard is intended chiefly to cover the technical provisions relating to the supply of amyl alcohol, and it does not include all the necessary provisions of a contract.

---

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for amyl alcohol.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given under 2 of IS : 82-1950* Methods of Test for Thinners and Solvents for Paints shall apply.

*Since revised.

3. GRADES

3.1 There shall be two grades of amyl alcohol, namely, Grade 1 and Grade 2.

3.1.1 *Grade* 1 — is intended for use in testing milk.

3.1.2 *Grade* 2 — is intended for use as an industrial solvent and especially as a thinner and solvent for paints.

4. REQUIREMENTS

4.1 Description

4.1.1 *For Grade* 1 — The material shall be a clear and colourless liquid and consists principally of *iso*amyl alcohol (*iso*butyl carbinol).

4.1.2 *For Grade* 2 — The material shall be clear and free from sediment and suspended matter.

4.2 The material shall also comply with the requirements given in Table I.

4.3 Special Requirement — When the material is required for use in pyrotechnic industry, it shall also comply with the requirements of moisture content, 0·4 percent, *Max* when tested by the method described in IS : 2362-1963*.

5. PACKING AND MARKING

5.1 Packing

5.1.1 Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in galvanized drums [conforming to IS : 2552-1963† Specification for Steel Drums (Galvanized and Ungalvanized)] or in any other container, subject to the provisions of the law in force in the country for the time being.

5.1.2 All containers in which the material is packed shall be dry and clean so that no impurities harmful to the end use of the material are introduced.

5.2 Marking

5.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

5.2.2 Each container shall have the caution label 'FLAMMABLE' together with the corresponding symbol for labelling of dangerous goods (*see* Fig. 3 of IS : 1260-1958‡ Code of Symbols for Labelling of Dangerous Goods).

NOTE 1 — Necessary safeguards against the risk arising from the storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion.

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*Determination of water by Karl Fischer method.

†Since revised.

‡Since revised and split into two parts.

TABLE I REQUIREMENTS FOR AMYL ALCOHOL

(*Clause* 4.2)

Sl No.	Characteristic	Requirement for		Method of Test	
		Grade 1	Grade 2	Ref to Appendix	Ref to Other Indian Standards
(1)	(2)	(3)	(4)	(5)	(6)
i)	Colour	Clear and colourless	Not greater than a freshly prepared solution of 0·003 0 g of potassium dichromate ($K_2 Cr_2 O_7$) in one litre of distilled water	—	5 of *IS : 82-1950
ii)	Water-content	—	Shall pass the test	A	—
iii)	Solubility in water	—	do	B	—
iv)	Specific gravity at 27/27°C	0·807 to 0·809	0·810 (*Max*)	—	6.3.3 of *IS : 82-1950
v)	Distillation range	Not less than 95 ml within 2 degrees in the range 128 to 132°C, the temperature being †corrected for a pressure of 760 mm of mercury	Not less than 95 ml between 125°C and 132°C, the temperature being †corrected for a pressure of 760 mm of mercury	—	Method B of P : 18 of ‡IS : 1448 (Part I)-1960
vi)	Residue on evaporation, g/100 ml, *Max*	—	0·01	—	8 of *IS : 82-1950
vii)	Acidity (as CH_3COOH), percent by weight, *Max*	—	0·012	C	—
viii)	Aldehyde content (as CH_3CHO), g/100 ml, *Max*	—	0·10	D	—
ix)	Furfural and other organic impurities	Shall pass the test	—	E	—
x)	Suitability for milk analysis	do	do	F	—
xi)	Hydrochloric acid test	do	—	G	—

*Methods of test for thinners and solvents for paints. (Since revised).

†In case the barometric pressure p deviates from 760 mm of mercury, use the correction 0·037 (p — 760) degree Centigrade for the distillation temperature.

‡Methods of test for petroleum and its products (*revised as* P : 18 in 1967).

NOTE 2 — Except when they are opened for the purpose of cleaning and rendering them free from amyl alcohol vapour, all empty tanks or other containers shall be kept securely closed unless they have been cleaned and freed from amyl alcohol vapour.

5.2.3 Each container shall be marked with:

a) name of the material;

b) manufacturer's name, initials or trade-mark, if any;

c) net, gross, and tare weight; and

d) month and year of manufacture.

5.2.4 The containers may also be marked suitably with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in Appendix H.

6.2 Number of Tests — Tests for the determination of all the characteristics specified shall be conducted on the composite sample.

6.3 Criteria for Conformity — The material shall be taken as conforming to this specification if the composite sample satisfies all the requirements prescribed.

7. TEST METHODS

7.1 Tests shall be conducted according to the methods as prescribed in various Indian Standards and the appendices to this standard. References to the relevant appendices and clauses of different standards are given in col 5 and 6 of Table I, respectively.

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water [*see* IS : 1070-1960 Specification for Water, Distilled Quality (*Revised*)] shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* I, *Item* (ii)]

TEST FOR WATER CONTENT

A-0. GENERAL

A-0.1 The material is empirically tested for water content by testing whether it develops any turbidity when mixed with petroleum hydrocarbon solvent in a specified ratio.

A-1. PROCEDURE

A-1.1 To 5 ml of the material contained in a 100-ml Nessler cylinder add 95 ml of petroleum hydrocarbon solvent 60/80 (conforming to *IS : 1745-1961 Specification for Petroleum Hydrocarbon Solvents) in 5-ml portions. Keep the Nessler cylinder with its contents at 27 ± 2°C during the test and shake the mixture well after each addition.

A-1.1.1 The material shall be regarded to have passed the test if no turbidity develops in the mixture.

APPENDIX B

[*Table* I, *Item* (iii)]

TEST FOR SOLUBILITY IN WATER

B-1. PROCEDURE

B-1.1 Measure 50 ml of the material in a 100-ml graduated measuring cylinder (conforming to IS : 878-1956 Specification for Graduated Measuring Cylinders) and add 50 ml of water. Adjust the temperature to 20°C, shake well and allow to stand. Observe the volume of the alcohol layer.

B-1.1.1 The material shall be taken to have passed the test if the volume of the alcohol layer is not less than 45 ml.

*Since revised.

APPENDIX C

[*Table* I, *Item* (vii)]

DETERMINATION OF ACIDITY

C-1. REAGENTS

C-1.1 Phenolphthalein Indicator — prepared by dissolving 0·1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

C-1.2 Standard Sodium Hydroxide Solution — 0·01 N.

C-2. PROCEDURE

C-2.1 Place 100 ml of water and a few pieces of clean porous porcelain in a 500-ml conical flask of resistance glass and boil gently for 5 minutes to eliminate carbon dioxide. Cool slightly and add 100 ml of the material. Boil gently for a further period of 5 minutes. At the end of this period, close the neck of the flask with a stopper carrying a soda-lime tube and allow to cool. When cold, remove the stopper, add 0·5 ml of phenolphthalein indicator and examine for alkalinity; if not alkaline, titrate with standard sodium hydroxide solution using a micro-burette.

C-3. CALCULATION

C-3.1 Calculate as follows:

$$\text{Acidity (as } CH_3COOH \text{), percent by weight} = \frac{6\,VN}{100\,S}$$

where

V = volume, in ml, of standard sodium hydroxide solution;

N = normality of standard sodium hydroxide solution; and

S = specific gravity of the material at the temperature of determination.

APPENDIX D

[*Table* I, *Item* (viii)]

DETERMINATION OF ALDEHYDE CONTENT

D-0. GENERAL

D-0.1 Hydroxylamine hydrochloride yields an oxime on reacting with the carbonyl group. The hydrochloric acid which is liberated is then titrated with standard sodium hydroxide solution.

D-1. APPARATUS

D-1.1 Stoppered Flasks — two, each of 250-ml capacity and identical in shape, size and colour.

D-2. REAGENTS

D-2.1 Sodium Hydroxide Solution — approximately 20 percent.

D-2.2 Solution of Hydroxylamine Hydrochloride — Dissolve 20 g of hydroxylamine hydrochloride in 100 ml of water.

D-2.3 Meta-phenylenediamine Hydrochloride

D-2.4 Aldehyde-Free Alcohol — Re-distil rectified spirit over solid caustic soda or caustic potash, add 2 to 3 g of meta-phenylenediamine hydrochloride per litre of rectified spirit. Digest at ordinary temperature for several days or under a reflux condenser on a steam-bath for several hours and distil slowly, rejecting the first 100 ml and the last 200 ml of the distillate.

D-2.5 Standard Sodium Hydroxide Solution — 0·1 N.

D-2.6 Bromophenol Blue Solution — Dissolve 0·1 g of bromophenol blue in 100 ml of rectified spirit [conforming to IS : 323-1959 Specification for Rectified Spirit (*Revised*)].

D-2.7 Hydroxylamine Reagent (Neutral Alcoholic Solution of Hydroxylamine) — Dilute 10 ml of the stock solution of hydroxylamine hydrochloride with 100 ml of aldehyde-free alcohol, add 2 ml of bromophenol blue solution and then add standard sodium hydroxide solution till the characteristic dichroic yellowish green colour is obtained.

D-3. PROCEDURE

D-3.1 Take 50 ml of the material in a flask, add 25 ml of water and 25 ml of hydroxylamine reagent. Allow to stand for 15 minutes. Titrate with standard sodium hydroxide solution until the same dichroic yellowish green tint appears as in a blank control made by treating 25 ml of distilled water only in a similar flask.

D-3.2 Calculation

Aldehyde content (as CH_3CHO), g/100 ml = $0{\cdot}088\ (V - v)\ N$

where

V = volume, in ml, of standard sodium hydroxide solution required for the titration;

v = volume, in ml, of standard sodium hydroxide solution required, if any, in the blank; and

N = normality of standard sodium hydroxide solution.

APPENDIX E

[*Table* I, *Item* (ix)]

TEST FOR FURFURAL AND OTHER ORGANIC IMPURITIES

E-0. GENERAL

E-0.1 It is based on halochromy developed by furfural and other organic impurities in the material with concentrated sulphuric acid.

E-1. PROCEDURE

E-1.1 Mix carefully 5 ml of concentrated sulphuric acid [conforming to IS : 266-1961 Specification for Sulphuric Acid (*Revised*)] with 5 ml of the material and then shake.

E-1.1.1 The material shall be taken to have passed the test if the solution does not show more than a yellow or light brown colour.

APPENDIX F

[*Table* I, *Item* (x)]

TEST FOR SUITABILITY FOR MILK ANALYSIS

F-0. GENERAL

F-0.1 The material is used in the Gerber Method for determination of fat in milk. Gerber test is carried out using water instead of milk. Butyrometer reading should report absence of any fat. Besides the same milk when examined by the Gerber Method using the material and by the Rose-Gottlieb Method should give a figure of fat content which should not differ by more than a stipulated limit.

F-1. PROCEDURE

F-1.1 Carry out the Gerber test according to the method prescribed under 4 of IS : 1224-1958 Determination of Fat in Whole Milk, Evaporated (Unsweetened) Milk, Separated Milk, Skim Milk, Buttermilk and Cream by the Gerber Method, using water instead of milk. The material shall be taken to have passed the test if butyrometer reading does not indicate any fatty layer on the top of the sulphuric acid.

F-1.2 When samples of milk are examined by the Gerber Method (*see* IS : 1224-1958), the results obtained shall not differ from those obtained by Rose-Gottlieb Method [*see* IS : 1479 (Part II) - 1961 Methods of Test for Dairy Industry : Part II Chemical Analysis of Milk] by more than 0·05 percent.

APPENDIX G

[*Table* I, *Item* (xi)]

HYDROCHLORIC ACID TEST

G-0. GENERAL

G-0.1 The material is mixed with an equal volume of concentrated hydrochloric acid and examined for complete miscibility. A millilitre of water. however, when added to the mixture separates the mixture into two layers,

G-1. PROCEDURE

G-1.1 Mix 10 ml of concentrated hydrochloric acid [conforming to IS : 265-1962 Specification for Hydrochloric Acid (*Revised*)] with 10 ml of the material and shake well.

G-1.1.1 The material shall be taken to have passed the test if a clear solution is formed and the solution separates into two layers on the addition of one millilitre of water.

APPENDIX H

(*Clause* 6.1)

SAMPLING OF AMYL ALCOHOL

H-1. GENERAL REQUIREMENTS OF SAMPLING

H-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

H-1.1 Samples shall be taken in a protected area with good ventilation. Keep samples away from flames.

H-1.2 The sampling instrument shall be clean and dry.

H-1.3 The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

H-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution

H-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass containers.

H-1.6 The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

H-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling and marked with the manufacturer's name or trade-mark, the month and year of manufacture of the material, the batch number (if available), and other details of sampling, such as the date of sampling and sampler's name.

H-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

H-2. SAMPLING INSTRUMENTS

H-2.0 The following forms of sampling instruments may be used:

a) Weighted sampling can for taking samples from various depths in large tanks, and

b) Sampling tube.

H-2.1 Weighted Sampling Can — of suitable capacity, 500- to 1 000-ml and of a such a weight as to sink readily in the material to be sampled. It has a long chain or cord attached to permit filling at any desired level (*see* Fig. 1). The metal used to weight the apparatus shall be fitted externally, as irregularities in the metal are likely to contaminate the sample if the weight is fitted internally.

H-2.2 Sampling Tube — It is made of metal or thick glass and is 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 2). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with the thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

H-2.2.1 For small container, the size of the sampling tube may be altered suitably.

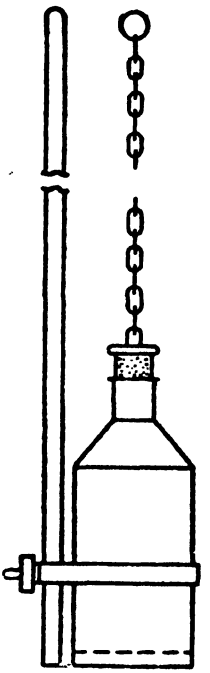

FIG. 1 WEIGHTED SAMPLING CAN

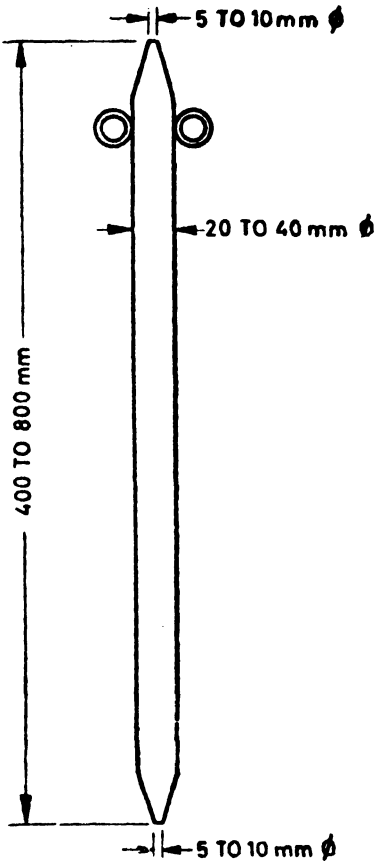

FIG. 2 SAMPLING TUBE

H-3. SCALE OF SAMPLING

H-3.1 Lot — In any consignment, all the containers of the same grade and size, and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different grades, batches of manufacture or of different sizes of containers, the containers belonging to the same grade or batch and size shall be grouped together and each such group shall constitute a separate lot.

H-3.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot

separately. For this purpose, 5 containers shall be selected at random from each lot.

NOTE — In the case of very small lots where the selection of 5 containers may be uneconomical, the number of containers to be selected and the method of judging the conformity of the lot to the requirements of the specification shall be as agreed to between the purchaser and the supplier.

H-3.3 The containers shall be selected at random and to ensure randomness of selection the following procedure is recommended for use:

Starting from any container in the lot, count them as 1, 2, ...up to r and so on, in one order, where r is the integral part of $N/5$ (N being the number of containers in the lot). Every rth container thus counted shall be withdrawn to constitute a sample till the required number of 5 containers is obtained.

H-4. PREPARATION OF TEST SAMPLES

H-4.1 From each of the containers selected according to **H-3.3**, a representative portion of the material sufficient for carrying out tests as prescribed in **4** shall be drawn with the help of the suitable sampling instrument (*see* **H-2**).

H-4.2 Out of these portions, equal quantity of material shall be taken and mixed thoroughly to form a composite sample of about 1 500 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

H-4.3 The composite sample shall be transferred to separate containers and shall be sealed and marked with full identification particulars given in **H-1.7**.

H-4.4 The referee test samples shall also bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two.

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